Effect of Hydroxypropyl Methylcellulose–Lipid Edible Composite Coatings on Plum (*cv. Autumn giant*) Quality During Storage

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ABSTRACT: 'Autumn Giant' plums were coated with edible hydroxypropyl methylcellulose-lipid composite coatings. The coatings consisted of beeswax or shellac, at 2 lipid content levels (20% and 60% dry basis). Weight loss of coated plums decreased as lipid content increased. No differences on weight loss were observed between uncoated and 20% lipid-coated plums, indicating that the natural waxes of plums are as effective as coatings having 20% lipid. Water-dipped plums experienced the highest weight loss. Fruit texture was not affected by coating after short-term storage at 20 °C. However, for prolonged storage at 20 °C, the coatings significantly reduced texture loss and internal breakdown compared to uncoated and water-dipped plums.

Keywords: autumn giant plums, edible composite coatings, beeswax, shellac, hydroxypropyl methylcellulose

Introduction

PLUMS ARE CLIMACTERIC FRUITS, WHICH ARE SUITABLE FOR COLD storage for a short period depending on the susceptibility to internal breakdown and loss in texture. The market life varies among cultivars from 1 to 8 wk and is affected by temperature management. Maximum market life is obtained when fruits are stored at approximately 0 °C and the minimum postharvest life occurs when stored around 5 °C (Crisosto and others 1999). Some cultivars have observed an improvement in postharvest life by the use of modified atmosphere (MA) or controlled atmosphere (CA) in combination with temperatures close to 0 °C. The major benefits of CA during storage are retention of fruit firmness and ground color. Conditions of 6% O_2 and 17% CO_2 are suggested for reduction of internal breakdown, but its effectiveness depends on cultivar, preharvest factors, market life, and storage time (Crisosto and others 2002).

Edible films and coatings can extend the shelf life and improve the quality of fruits and vegetables by creating a modified atmosphere inside the fruit due to the barrier to O_2 and CO_2. In addition, edible coatings can reduce weight loss, carry food ingredients, and/or improve the mechanical integrity or handling characteristics of the product (Krochta 1997). The principal disadvantage, however, is the development of off-flavors if the inhibition to O_2 and CO_2 exchange results in anaerobic respiration. The barrier properties depend on the chemical composition and structure of the film-forming polymer and the conditions of storage. Many studies have focused on elucidating how composition, preparation, and storage conditions affect the properties of standing-alone films and reviews can be found (Guilbert 1986; Kester and Fennema 1986; Krochta and others 1994; Krochta and De Mulder-Johnston 1997; Debeaufort and others 1998). This knowledge is required to better understand the transfer mechanisms of gases and solutes through these edible materials. Currently, there are several polysaccharide-based coatings commercially available for postharvest use. However, the lack of knowledge about composition on many commercially available coatings makes it difficult to predict their performance on fruit quality. Therefore, there is a need to study the effect of coating composition on postharvest quality of specific commodities.

A few papers have been found in the literature where coatings have been applied to plums, showing some improvement in storage life and fruit quality (Dinamarca and others 1989; Basiouny and Baldwin 1997). The effect of edible coatings, creating a modified atmosphere inside the fruit due to the barrier to gases O_2 and CO_2, is to extend the market life and improve quality by reducing the internal breakdown and loss in texture. This investigation aims to characterize the effect of lipid type and amount of hydroxypropyl methylcellulose (HPMC)–lipid composite coatings on postharvest quality of plums cv. Autumn Giant.

Materials and Methods

Materials

Hydroxypropyl methylcellulose (HPMC) (Methocel E15) was supplied by Dow Chemical Co. (Midland, Mich., U.S.A.). Refined beeswax (BW) (grade 1) and dewaxed-decolourised flake shellac (acid nr 69.7) were purchased from Brillocera, S.A (Valencia, Spain). Stearic acid, glycerol, and ammonium hydroxide (30%) were from Panreac Química, S.A. (Barcelona, Spain).

Coating formulation

Emulsion coatings consisted of HPMC and beeswax (BW), or shellac, as the hydrophilic and lipidic phase, respectively, suspended in water. To make the emulsion coatings, 5% HPMC was dispersed in hot water at 80 °C. Next, stearic acid and glycerol were added as emulsifier and plasticizer, respectively. The HPMC-plasticizer phase consisted of 2 parts HPMC to 1 part glycerol (dry basis), and this ratio was kept constant throughout the study. Lipid (BW or shellac)-stearic acid ratio was also kept constant and consisted of 5 parts lipid to 1 part fatty acid (dry basis). Either the BW or shellac were added to the HPMC-stearic acid-glycerol mixture at 2 different levels (20% and 60% dry basis). To help melting of the BW and shellac, solutions were previously heated to 10-to-20 °C above the melting point of the lipids, so they melted immediately. Since shellac is an alkali-soluble resin, the pH was increased by adding 1% NH_3. Once the lipids were melted, samples were homogenized with a high-shear probe mixer (PolyTron, Model PT 2100; Kinematica AG Inc., Lucerne, Switzerland) for 4 min at 30000 rpm. After ho-

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mogenization, cold water was added to bring the emulsions to 4% total solids content. Further cooling was achieved by placing the emulsions in an ice bath to bring them to less than 30 °C. Agitation was continued for approximately 20 min after reaching this temperature to ensure complete hydration of the HPMC. Composition of the emulsion coatings on at dry bases is shown in Table 1.

Table 1—Coating composition on a dry basis (%)

Components	20% lipid content coating	60% lipid content coating
HPMC	50.7	18.7
Lipid	20.0	60.0
Stearic acid	4.0	12.0
Glycerol	25.3	9.3

HPMC = hydroxypropyl methylcellulose
Coating formulations were prepared by adding 1% NH_3 (wet basis).
Coating formulations were prepared at 4% solid content.

Sample preparation-coating application

'Autumn Giant' plums were harvested from a local grove in Valencia (Spain) and transported the same day to the research laboratory. The fruits were stored 1 d at 5 °C and 90 to 95% RH in the presence of Fruitfog-P (Fomesa S.A, Valencia, Spain), which consisted of 25% orthophenylphenol, in order to disinfect the fruit. After 1 d of storage, the fruits were selected for size, color, and absence of physical damage. Plums were randomly divided into 6 groups, which corresponded to 4 coating treatments, 1 water-dipped treatment, and 1 uncoated-untreated control. Fruits were dip-coated by immersion in the coating solutions for 90 s, drained of excess coating, and air-dried at room temperature with a fan which had an airflow of 7500 m^3/h.

After coating, the fruits were stored up to 6 wk at 1 °C and 85 ± 5% RH, followed by 3 d at 6 °C simulating transport conditions and 1 additional d at 20 °C, simulating retail handling conditions. Storage time at 20 °C was also prolonged up to 20 or 25 d for samples previously stored for 4, 5, and 6 wk at 1 °C, followed by 3 d at 6 °C.

Weight loss

Lots of 30 fruits per treatment were used to measure weight loss. The same fruits were weighed at the beginning of the experiment and at the end of each storage period. The results were expressed as the percentage loss of initial weight.

Fruit firmness

Resistance to penetration force was measured in 20 plums from each treatment and 2 tests per fruit, one on each of the opposite cheeks. Previous to the measurement, a disk of the skin of about 2 cm in dia was removed, so measurements were made from the flesh. Firmness was determined at the end of each storage period using an Instron Universal Testing Machine (Model 4301; Instron Corp., Canton, Mass., U.S.A.) using a plunger of 11 mm dia, by recording the force in newtons (N) required to penetrate the fruit.

Ethylene production

Three replicates of 4 fruits each were used to determine C_2H_4 production at the end of the storage. Samples were weighed and placed in sealed containers of known volume. The accumulation of C_2H_4 in the atmosphere was measured at 20 °C over a period of 20 h. To avoid ethylene inhibition due to CO_2 accumulation, 25 mL of 1N KOH was included in the containers. The gas sample (1 mL) was then injected into a Perkin Elmer gas chromatograph (Model 2000; Norwalk, Conn., U.S.A.) fitted with a Porapack QS 80/100 (1.2 m × 0.32 cm i.d.) column. Temperatures were 75 °C, 175 °C, and 300 °C, respectively, for the column, injector, and flame ionization detector. Helium was used as carrier gas at a flow rate of 30 mL/min. Peak areas obtained from standard gas mixtures were determined before and after analysis of samples and results were expressed as $\mu L\ C_2H_4$ / kg h.

Ethanol content

Ethanol concentration in juice was determined by head-space gas chromatography according to the method described by Ke and Kader (1990). Ten fruits each in 3 replicates per treatment were analyzed. Five mL of juice was transferred to 10-mL vials with crimp-top caps and TFE/silicone septum seals. Ethanol was analyzed using a Perkin Elmer gas chromatograph (Model 2000) with a flame ionization detector and a 1.2 m × 0.32 cm (i.d.) Porapack QS 80/100 column. The injector was set at 175 °C, the column at 150 °C, the detector at 200 °C, and the carrier gas at 9.1 psi. A 1-mL sample of the head-space was withdrawn, from vials previously equilibrated in a water bath at 20 °C for 1 h, followed by 15 min at 60 °C, to reach equilibrium in the head-space, and injected in the GC. Ethanol was identified by comparison of retention times with standards. Results were expressed as mg/100 mL juice.

Deterioration index

The physiological disorders reported in the literature for plums include mealiness, flesh browning, black pit cavity, flesh translucency, lack of juiciness, and red pigment accumulation. These physiological disorders are reported as internal breakdown or chilling injury (Crisosto and others 1999). For the visual rating of internal breakdown, fruit were cut in half and evaluated on the mesocarp and the area around the pit. Eighty fruits per treatment were inspected for physiological disorders at the end of each storage period. The different degrees of physiological disorders were rated as 0 = none, 1 = light, 2 = moderate, and 3 = severe. Light was considered when less than 10% of the flesh area was affected and severe when more than 25% of the flesh area was affected. Results were converted to an average index.

Statistical analysis

Statistical analysis was performed using STATGRAPHICS Plus 2.1 (Manugistics, Inc., Rockville, Md., U.S.A.). Specific differences between means were determined by least significant difference (LSD). Significance of differences was defined at $p \le 0.05$.

Results and Discussion

Plum weight loss

Figure 1 shows changes of weight loss of coated, water-dipped, and uncoated plums with storage time. Weight loss of coated plums decreased as lipid content of coating formulations increased ($p \le 0.05$). These differences, even though significantly different, were small when plums were stored up to 3 wk at 1 °C, followed by storage at 6 °C and 20 °C. After 4 wk of storage at 1 °C, an increase in shellac content seems to be more effective reducing weight loss than an increase in BW content. At similar lipid content, lipid type had little effect on weight loss when plums were stored up to 3 wk at 1 °C, followed by storage at 6 °C and 20 °C. After 4 wk of storage at 1 °C, 20% BW-based coatings were more effective reducing weight loss than 20% shellac-based coatings, whereas 60% shellac-based coatings provide higher moisture barrier than 60% BW-based coatings ($p \le 0.05$). When similar coatings were applied to 'Fortune' mandarins, coating performance on weight loss was not generally affected by lipid type at 20% lipid content; however, at 60% lipid content, BW-based coatings provided lower weight loss than shellac-based coatings (Perez-Gago and others 2002).

No differences on weight loss were observed between uncoated-untreated and 20% lipid-coated plums. However, when plums were water-dipped and no coating was applied, weight loss was significantly higher than with uncoated and coated plums, probably due to the partial removal of the wax layer of the cuticle. Plums are naturally covered by a continuous wax layer that protects the fruit against environmental stresses and contributes as a barrier to gas diffusion. In the present experiment, the results indicate that the diffusion resistance of the natural wax layer exerted a moisture barrier comparable to the coatings containing 20% lipid, which indicates that in order to improve moisture barrier of 'Autumn Giant' plums, coatings containing more than 20% lipid need to be applied. Storey and Price (1999) studied the microstructure of the skin of 'd'Agen' plums, observing that the epicuticular wax was a closely packed granular structure overlying a more amorphous layer, which was more than 5 μm thick. In addition, these authors observed very few cuticular fractures over the surface of 'd'Agen' plums apart from those associated with stoma, contrary to the epicuticular wax of other fruits, such as apple, pear, and nectarines, which frequently fracture during fruit growth (Knuth and Stosser 1987; Kovás and others 1994; Nguyen-The 1991). This wax layer of d'Angen plums was supposed to be responsible for the high resistance to water movement across the cuticle during drying (Price and others 2000).

Fruit firmness

Fruit firmness was measured after cold storage at 1 °C, followed by 3 d at 6 °C plus 1 d at 20 °C. In general, regardless of the behavior of coating type on firmness, application of edible coatings to fruits and vegetables reduces texture loss during storage. In this experiment, fruit firmness was significantly lower after 5 and 6 wk of storage at 1 °C compared to shortest storage periods ($p \leq 0.05$). However, coating application did not improve plum firmness compared to water-dipped and uncoated plums at any of the storage conditions, which could be due to the cold temperatures used during storage and the short period of time at 20 °C (data not shown). For this reason, we decided to study the effect of the coatings under prolonged storage at 20 °C. Samples stored 4, 5, and 6 wk at 1 °C, followed by 3 d at 6 °C, were kept between 20 and 25 d at 20 °C. Under these storage conditions, coating application significantly improved firmness compared to water-dipped and uncoated plums (Figure 2). At these storage conditions, uncoated and water-dipped plums were very soft, reaching levels considered unmarketable, whereas coated plums showed significantly higher firmness levels ($p \leq 0.05$).

The effect of the coatings on fruit firmness could be attributed to the beneficial effects of atmospheres with low O_2 and/or high CO_2 content on reducing softening. Ke and others (1991) have reported the effect of low-O_2 atmospheres on postharvest physiology and quality attributes of 'Angeleno' plums stored at 5 °C and at 10 °C for 25 and 35 d in air and in 0.25% or 0.02% O_2 atmosphere. Similarly to our results, these authors reported that for plums stored at 5 °C, flesh firmness did not show any significant differences in any of the storage conditions. However, when the storage temperature was increased to 10 °C, the samples stored under low-O_2 atmospheres presented higher flesh firmness than the samples stored in atmospheric conditions.

Ethanol content

The resistance to gas diffusion created by coating application to fresh fruit can reduce respiration rate by creating a modified atmosphere inside the fruit. If the gas barrier created by the coating is too high, the coatings can induce an increase in the amount of some internal volatiles associated with anaerobic conditions. In this work, the results confirm the creation of a modified atmosphere, as can be seen by the lower ethanol accumulation during storage in uncoated and water-dipped fruits than in coated fruit (Figure 3). Under all storage conditions, 20% lipid content coatings gave higher ethanol levels than 60% lipid content coatings, which indicates that 20% lipid content coatings provide more of a gas barrier between the fruit and the surrounding atmosphere. Similar results were found in a previous experiment when similar coatings were applied to 'Fortune' mandarins (Perez-Gago and others 2002). In the previous work, differences were attributed to the low oxygen permeability of HPMC (20% lipid-content coatings have higher levels of HPMC than 60% lipid-content coatings) and/or the differences in coating thickness between 20% and 60% lipid content coatings attributed

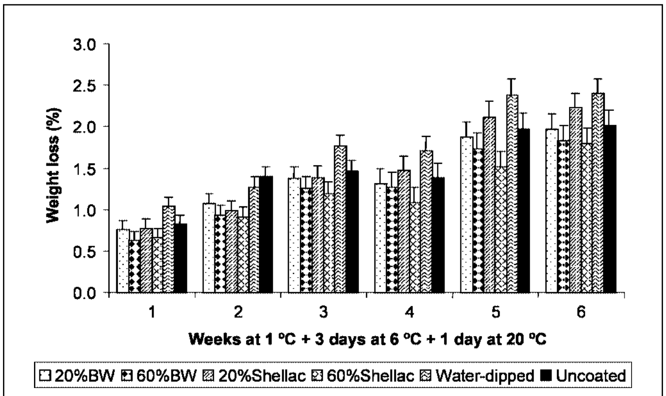

Figure 1—% Weight loss of 'Autumn Giant' plums coated with HPMC:lipid composite coatings as affected by lipid type and amount. Error bars within each storage time indicate LSD values at $p \leq 0.05$ (n = 30).

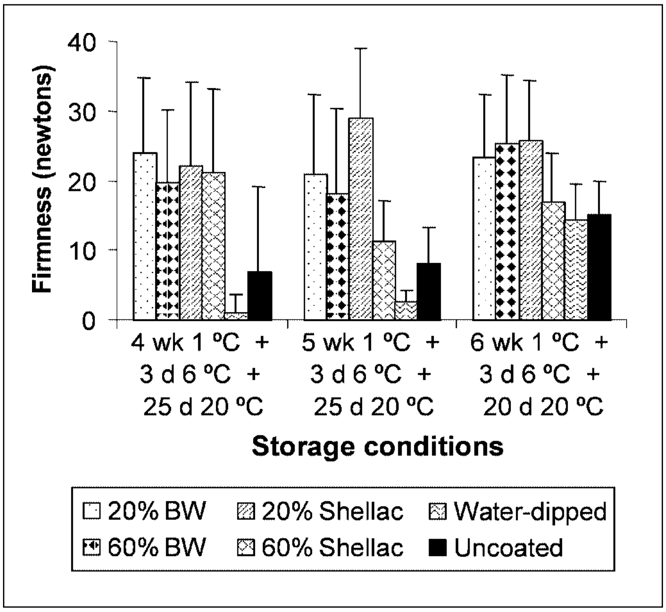

Figure 2—Firmness of 'Autumn Giant' plums coated with HPMC:lipid composite coatings after prolonged storage at 20 °C. Error bars indicate standard deviations (n = 20).

to differences in emulsion viscosity (20% lipid-content emulsions had higher viscosity than 60% lipid-content emulsions).

Shellac-based coatings generally have been reported as inducing higher ethanol contents in citrus fruit than wax coatings, due to the higher gas barrier that this resin provides compared to waxes (Hagenmaier and Baker 1994; Baldwin and others 1995; Hagenmaier 2002). In our experiment, 20% shellac-based coatings exhibited the highest ethanol levels. At 60% lipid content, shellac-based coatings gave higher ethanol levels than BW-based coatings only in plums stored 4 wk at 1 °C.

Ethylene efflux

Ethylene is a powerful plant hormone that has many effects on the growth, development, and storage life of many fruits (Saltveit 1999). Ethylene production rates are reduced by reduced O_2 levels, and elevated CO_2 levels around the commodity (Kader 1992). Therefore, the application of edible coatings could modify ethylene production in the commodity if the coating were able to provide a gas barrier between the fruit and the surrounding atmosphere. Table 2 shows the ethylene values of coated, water-dipped, and uncoated plums with storage time. A maximum ethylene peak was reached after 2 wk of storage at 1 °C, followed by 3 d at 6 °C plus 1 d at 20 °C. At this storage time, coating application and composition did not affect ethylene efflux of the fruit. However, there was initiation of ethylene production in water-dipped plums after 1 wk of storage at 1 °C, whereas uncoated and coated plums did not show ethylene synthesis (values not detected by gas chromatography). After 3, 4, and 6 wk of storage at 1 °C, followed by storage at 6 °C and 20 °C, water-dipped and uncoated samples showed higher ethylene levels than most of the coated samples ($p \leq 0.05$). However, these differences were very small. The internal modified atmosphere created by the coatings, and/or the stress induced during washing could be the reason for the low and high ethylene efflux in coated and water-dipped fruit, respectively.

In general, there was no observed difference in ethylene production depending on coating type. Results reported in the literature show that the effect of coating application on ethylene synthesis depends on fruit commodity and type of coating. It has been found that Prolong (a sucrose polyester of fatty acids and sodium salts of carboxymethyl cellulose) did not affect ethylene synthesis of Tsugaru apples (Lee and others 1986), but reduced ethylene production of bananas (Quantick and others 1996). Reduction in ethylene production has also been reported for avocados coated with NatureSeal (Bender and others 1993), tomatoes coated with chitosan (El-Ghaouth and others 1992) and Elstar apples coated with sucrose fatty acid ester (Xuan and others 2000). However, ethylene production was not modified in Red Delicious apples and celery sticks coated with caseinate-acetylated monoglyceride edible coatings (Avena-Bustillo and others 1997).

Table 2—Ethylene efflux of coated and uncoated '*Autumn Giant*' plums (μL C_2H_4/kg h)

Coating formulation	Storage time (wk at 1 °C + 3 d at 6 °C + 1 d at 20 °C) 1 wk	2 wk	3 wk	4 wk	5 wk	6 wk
20% BW	nd	1.98 a	0.05 b	0.03 a	0.03 a	0.01 a
60% BW	nd	1.88 a	0.05 b	0.04 ab	0.01 a	0.01 a
20% Shellac	nd	1.84 a	0.06 b	0.03 a	0.05 a	0.02 a
60% Shellac	nd	1.97 a	0.03 a	0.05 b	0.05 a	0.03 a
Water-dipped	1.25	1.85 a	0.09 c	0.05 b	0.04 a	0.06 ab
Uncoated	nd	1.73 a	0.09 c	0.06 b	0.03 a	0.12 b

Means followed by the same letter within each storage time are not significantly different at $p \leq 0.05$
nd = none detected

Physiological disorders

Plum fruit are highly perishable, and some cultivars develop mealiness, flesh browning, black pit cavity, flesh translucency, red pigment accumulation, failure to ripen, and lose flavor after prolonged cold storage and/or after ripening at room temperature (Crisosto and others 1999). These symptoms are reported as internal breakdown (IB) or chilling injury (Mitchell and Kader 1989, Crisosto and others 1999). In this experiment, mealiness and flesh browning were the major IB symptoms in 'Autumn Giant' plums.

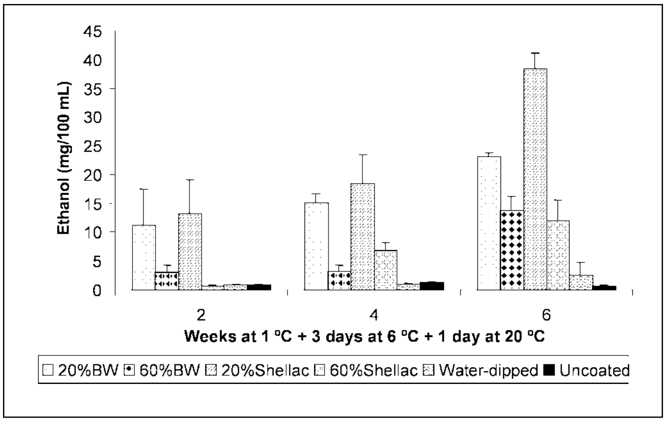

Figure 3—Ethanol content in juice of 'Autumn Giant' plums coated with HPMC:lipid composite coatings as affected by lipid type and amount. Ethanol contents are mean values of 3 replicates (10 fruits each) and 3 repeated observation per replicate. Error bars indicate standard deviations.

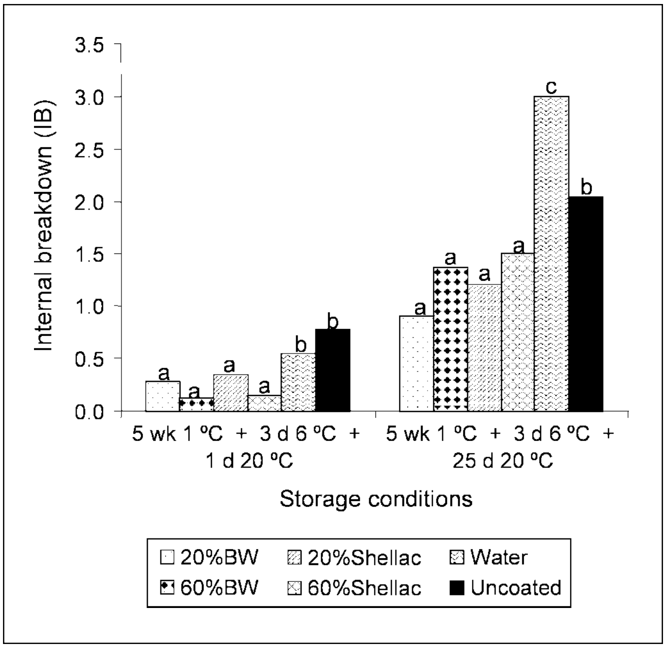

Figure 4—Internal breakdown (IB) of 'Autumn Giant' plums coated with HPMC:lipid composite coatings. IB ranked from 0 (none) to 3 (severe). Means within each storage time with the same letter are not significantly different ($p \leq 0.05$).

Figure 4 shows the IB for coated and uncoated plums stored 5 wk at 1 °C, followed by 3 d at 6 °C and 1 or 25 d at 20 °C. The range of IB depended on the treatment and the storage conditions. When the plums were stored for 1 d at 20 °C they showed low IB. At this storage condition, water-dipped and uncoated plums had higher IB than coated plums, but the level could be considered negligible. When storage time at 20 °C was prolonged to 25 d, IB increased. The application of the coatings significantly reduced IB compared to water-dipped (severe IB) and uncoated (high IB) plums. Similar behavior was obtained when plums had been previously stored 4 or 6 wk at 1 °C, followed by 3 d at 6 °C and storage at 20 °C. These results correlate with the results found on fruit texture, where plum quality was improved by coating application for prolonged storage at 20 °C.

Conclusion

LIPID TYPE AND CONTENT HAD AN EFFECT ON WEIGHT LOSS AND ethanol build-up, which indicates their effectiveness as moisture and gas barrier. In general, 20% BW-based coatings were more effective reducing weight loss than 20% shellac-based coatings, whereas, 60% shellac-based coatings seemed to provide higher moisture barrier than 60% BW-based coatings. No differences on weight loss were observed between uncoated and 20% lipid-coated plums, whereas water-dipped plums had significantly higher weight loss than uncoated and coated plums. These results indicate that, in order to improve moisture barrier of 'Autumn Giant' plums, coatings containing more than 20% lipid need to be applied. The application of these edible coatings improved plum quality for prolonged storage at 20 °C, by the retention of firmness and the reduction in internal disorders, which could be explained by the gas barrier provided by the coatings creating a modified atmosphere in the fruit.

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MS 20020668 Submitted 11/22/02, Revised 1/6/03, Accepted 1/24/03, Received 1/27/03

This work was funded by the Spanish Agriculture Dept. through the project AGL 2002-00560. The authors also thank Dow Chemical Co. and Cat-Preymer for supplying samples of hydroxypropyl methylcellulose and technical support.

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